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Study of hyperfine interactions in spinel cobalt ferrite CoFe_2O_4 doped with Hf, Lu, and Yb using Mössbauer spectroscopy and perturbed γ - γ angular correlation

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Abstract

We studied hyperfine interactions (HFI) in spinel cobalt ferrite (ferrospinel) CoFe_2O_4 doped with Lu, Yb, and Hf (1 wt. %) by Mössbauer spectroscopy (MS) on ^{57}Fe nuclei. The interactions indicate the presence ($\text{CoFe}_2\text{O}_4\text{:Lu} - 11\%$, $\text{CoFe}_2\text{O}_4\text{:Yb} - 23.4\%$) and the absence ($\text{CoFe}_2\text{O}_4\text{:Hf}$) of additional phases. The study revealed a significant change in the HFI parameters on ^{57}Fe nuclei in the octahedral sites in ferrospinel doped with Hf, Lu, and Yb. However, the relative influence of the impurity on ^{57}Fe nuclei in the tetrahedral sites was insignificant.

The parameters of hyperfine fields on ^{172}Yb nuclei in cobalt ferrite were obtained by the method of perturbed γ - γ angular correlations (PAC). The ^{172}Yb ions were introduced into the sample using two methods: by adding ^{172}Hf and ^{172}Lu isotopes. A significant difference in the HFI parameters for these two cases was revealed. The local environment of ^{172}Yb ions appears to be different in the two variants of isotope introduction into the sample (Hf or Lu). The difference in the HFI parameters persisted in the temperature range of 300 - 1000 K.

It was determined that the different effects of Hf and Lu on the parameters (electric field gradient, magnetic field, and isomer shift) of the HFI in the sample are revealed by both MS and PAC methods, irrespective of the amount of the dopant. According to the MS data, Hf and Lu do not lead to significant changes in the HFI parameters in the tetrahedral sites ("Sextet 1"), but have a significantly different effect on the same parameters in the octahedral sites ("Sextet 2"). A similar pattern was observed using the PAC method: replacing Hf with Lu did not reveal any changes in the HFI parameters in one of the sites (the octahedral site), but indicated a significant change in the other site.

Keywords: Mössbauer spectroscopy, Perturbed correlations, Spinels, Oxides, Ferrites

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1. Introduction

Ferrites are mixed metal oxides with the general formula MFe_2O_4 , where the metal (M) may be represented by Fe, Ni, Co, Zn, etc. They have a number of properties (high resistivity, high magnetic permeability, and high penetration depth of microwave field) that contribute to their application in various fields [1–9]. Cobalt ferrite (CoFe_2O_4) is one of the most widely used magnetic materials. In modern technologies, ferrites are often doped, because even small ($\approx 1\%$) amounts of additives significantly change their properties. Currently, rare earth elements (RE) are considered as dopants [6–15]. In the case of modification with RE^{3+} ions, taking into account their typical oxidation state of +3, we can expect them to replace Fe^{3+} [11–15]. On the other hand, considering that the ionic radius of the RE is larger than the ionic radius of Fe^{3+} , such substitution is difficult, and an additional phase may be formed [14]. Ferromagnetic spinel semiconductors CdCr_2Se_4 and CdCr_2S_4 were studied by the PAC method at the ISOLDE on-line separator (CERN) [16]. But in that work, ferrites were not studied, and besides the introduced isotopes (^{111}In , $^{111\text{m}}\text{Cd}$, ^{111}Ag , ^{117}Cd , and $^{199\text{m}}\text{Hg}$) were not rare earth elements. In work [17], non-rare earth impurities of CoFe_2O_4 were studied by Mössbauer spectroscopy.

The aim of this work was to study the hyperfine interactions in spinel cobalt ferrite (ferrospinel) CoFe_2O_4 doped with Lu, Yb, and Hf by Mössbauer spectroscopy (MS) and perturbed angular correlation (PAC) methods.

2. Experimental

2.1. Synthesis of samples

For the study, samples of cobalt ferrite CoFe_2O_4 were obtained by adding dopants during the synthesis. In the first case (the MS method), natural Yb, Lu, and Hf were added in the amount

of 1 wt. % of the total weight of the sample. For the PAC measurements, CoFe_2O_4 was produced with the addition of radionuclides during the synthesis.

Ferrites were synthesized from mixed aqueous solutions of Fe and Co salts with a molar ratio of 2:1 by precipitation with 1 M NaOH solution to the pH of 11–12. To modify the samples with stable Yb, Lu, and Hf or radionuclides, solutions of Yb, Lu, or Hf nitrates or solutions of ^{172}Lu or ^{172}Hf radionuclides were added to the initial aqueous solutions of Fe(III) and M(II) ($\text{M} = \text{Co}$) salts. The procedure was described in detail in [13, 14, 15]. The preparation of radionuclides was described in [18]. The final washing of the precipitate was carried out with a 50/50 (v/v) water/acetone mixture. The washed samples were dried at 80°C overnight and then heat treated at 750°C in air for 5 hours. The obtained mixed metal oxides based on CoFe_2O_4 were labelled as CFO.

2.2. Mössbauer spectroscopy

The Mössbauer spectra were measured on a MS-1104Em spectrometer [20] in the sample mass range from 1 to 5 mg. The spectrometer was operated in transmission geometry at room temperature. The ^{57}Co radiation source was modulated in a mode in which the dependence of the Doppler velocity over time has a triangular shape. A ^{57}Co source in a Cr matrix with an activity of 30 mCi produced by Ritverc JSC was used as a resonant source of γ -quanta. A scintillation detector based on a NaI scintillator was used. It was calibrated against the metallic α -Fe. The speed was 12.11 mm/s, the triangular velocity profile of the absorber was used to record the Mössbauer spectra at forward and reverse movement. The Mössbauer spectra were approximated by Lorentzian lines according to the χ^2 criterion.

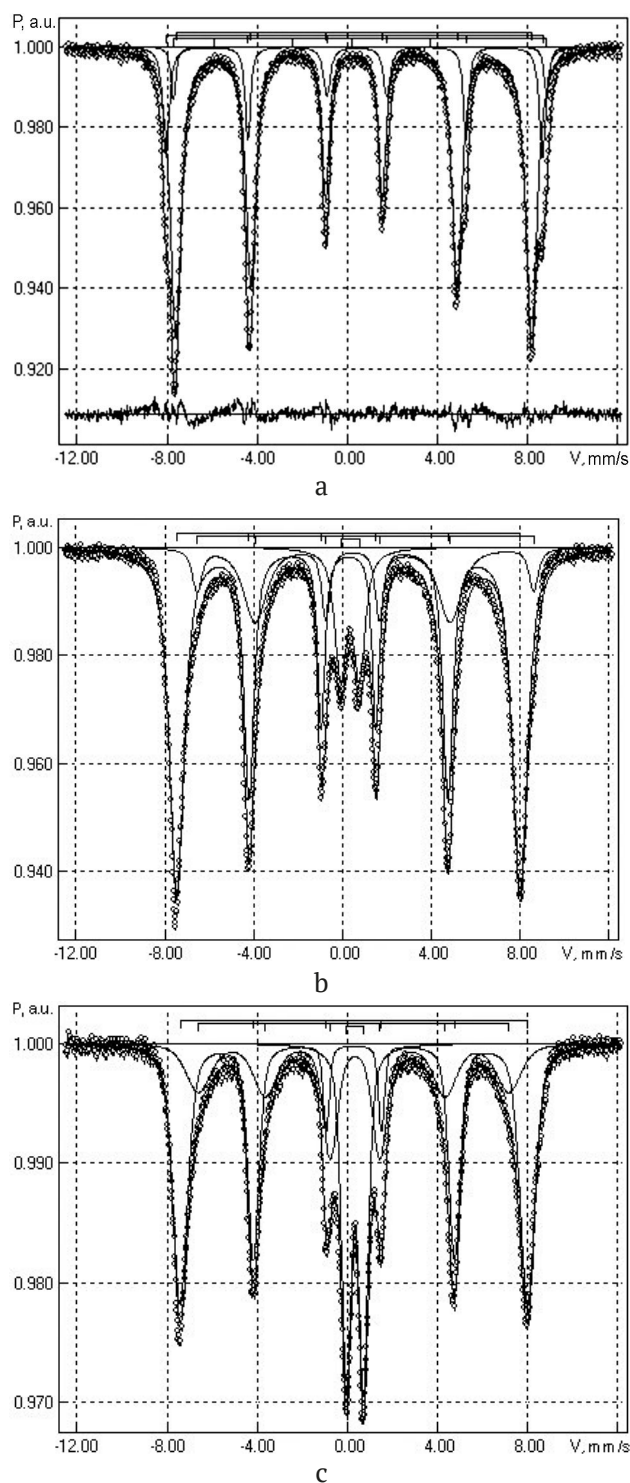


Fig. 2. Mössbauer spectra for ^{57}Fe in samples: a) $\text{CoFe}_2\text{O}_4\text{:Hf}$; b) $\text{CoFe}_2\text{O}_4\text{:Lu}$, and c) $\text{CoFe}_2\text{O}_4\text{:Yb}$, measured at 298 K

a negligible influence in the A sites.

Studying the ^{57}Fe quadrupole state in CoFe_2O_4 (1 wt. %) Hf, Lu, and Yb ferrites (Fig. 4) also provided important results. We observed a large ^{57}Fe quadrupole splitting in CoFe_2O_4 (1 wt. %)

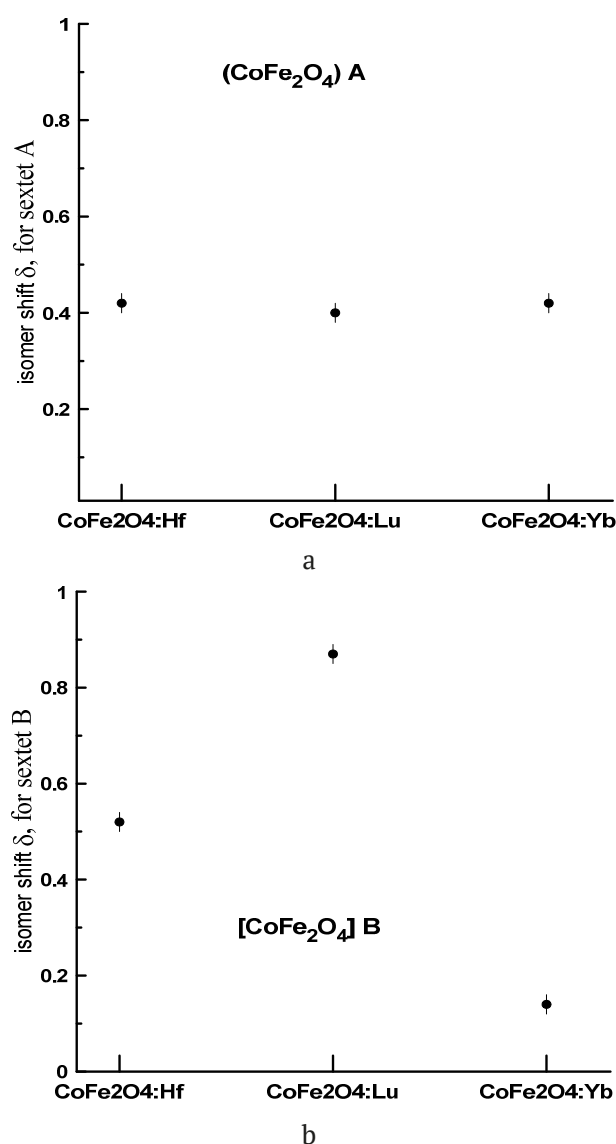


Fig. 3. The ^{57}Fe isomer shift in CoFe_2O_4 (1 wt. %) Lu, Yb, and Hf in the sites: a) $(\text{CoFe}_2\text{O}_4)\text{A}$ and b) $[\text{CoFe}_2\text{O}_4]\text{B}$

Lu relative to ferrites containing Yb and Hf. In addition, the ^{57}Fe quadrupole splitting appears only in the $[\text{CoFe}_2\text{O}_4]\text{B}$ sites.

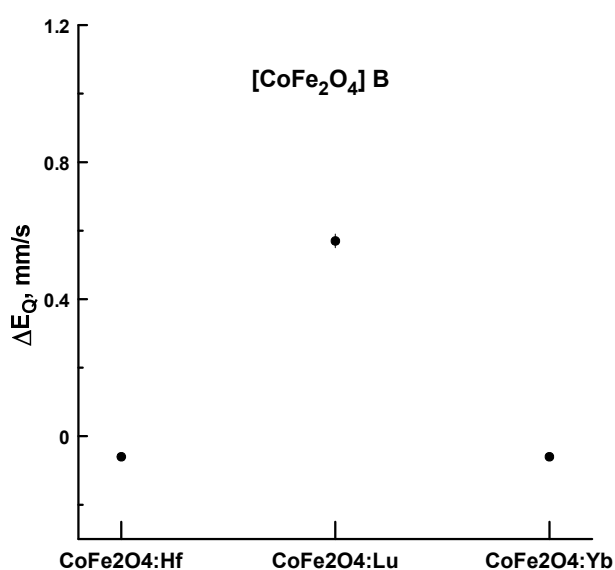
The results for the ^{57}Fe hyperfine magnetic field in CoFe_2O_4 (1 wt. %) Hf, Lu, and Yb are shown in Fig. 5a, b. We observed a significant change in the ^{57}Fe hyperfine field in the $[\text{CoFe}_2\text{O}_4]\text{B}$ site depending on the impurity element. The influence of the impurity element on ^{57}Fe in the $[\text{CoFe}_2\text{O}_4]\text{A}$ sites was negligible.

3.2. Studies of CoFe_2O_4 ferrites by the PAC method

For the PAC measurements on ^{172}Yb nuclei, we can add either ^{172}Lu or ^{172}Hf radioactive

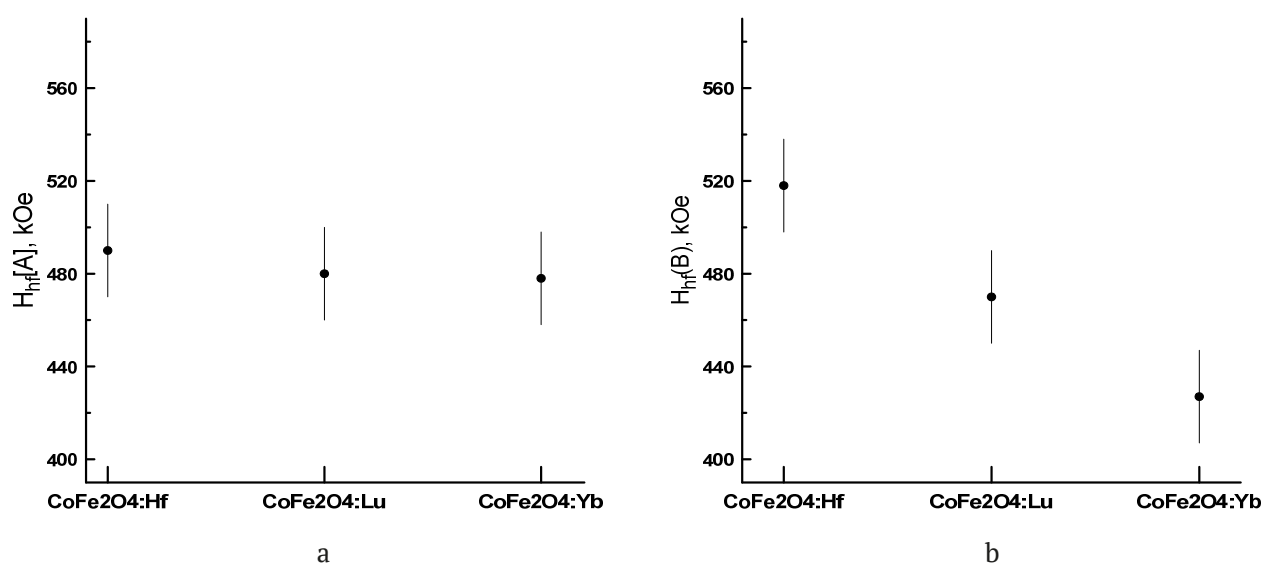
Table 1. Mössbauer parameters of CoFe_2O_4 samples. δ is the isomer shift, ΔE_Q^a is the quadrupole splitting, G is the line width, H is the Fe magnetic field, and A is the area under the spectrum

Sample	Sextet in the spectrum	δ (mm/s)	ΔE_Q^a (mm/s)	G (mm/s)	H (kOe)	A (%)	Component
CoFe_2O_4 +1 % Hf	«Sextet_1»	0.42	-0.00	0.56	490	77.40	$(\text{CoFe}_2\text{O}_4)_A$
	«Sextet_2»	0.52	-0.06	0.44	518	22.60	$[\text{CoFe}_2\text{O}_4]_B$
CoFe_2O_4 +1 % Lu	«Sextet_1»	0.40	-0.00	0.80	480	68.50	$(\text{CoFe}_2\text{O}_4)_A$
	«Sextet_2»	0.87	0.57	0.50	470	20.46	$[\text{CoFe}_2\text{O}_4]_B$
	«Doublet_1»	0.47	0.84	0.63		11.04	CoFe
CoFe_2O_4 +1 % Yb	«Sextet_1»	0.42	-0.00	0.79	478	48.83	$(\text{CoFe}_2\text{O}_4)_A$
	«Sextet_2»	0.14	-0.06	1.29	427	27.87	$[\text{CoFe}_2\text{O}_4]_B$
	«Doublet_1»	0.14	0.74	0.51		23.39	CoFe

**Fig. 4.** The ^{57}Fe quadrupole splitting in CoFe_2O_4 (1 wt. %) Hf, Lu, and Yb ferrites

isotope to the sample. It should be noted that the radioactive isotopes were added without a carrier. An important assumption, apparently quite obvious, is that the position of the probe nucleus in the sample matrix coincides with the position of the parent nucleus.

The results at room temperature (considerably below the Curie temperature) showed that the CoFe_2O_4 ferrite (CFO) with incorporated ^{172}Hf (Fig. 6, above) provided two states of ^{172}Yb , characterized by two magnetic fields $B_{\text{hf1}} = 14(1)$ T and $B_{\text{hf2}} = 17(1)$ T with a population ratio of $\sim 2:1$. In the case of incorporated ^{172}Lu (Fig. 6, below), the processing of the spectrum indicated the presence of a single magnetic field $B_{\text{hf}} = 18(1)$ T. This significant difference can be explained by the assumption that at room temperature ^{172}Lu is introduced preferentially into only one type of crystal lattice sites.

**Fig. 5.** The ^{57}Fe hyperfine magnetic field in CoFe_2O_4 (1 wt. %) Lu, Yb, and Hf ferrites in the sites: a) $(\text{CoFe}_2\text{O}_4)_A$ and b) $[\text{CoFe}_2\text{O}_4]_B$

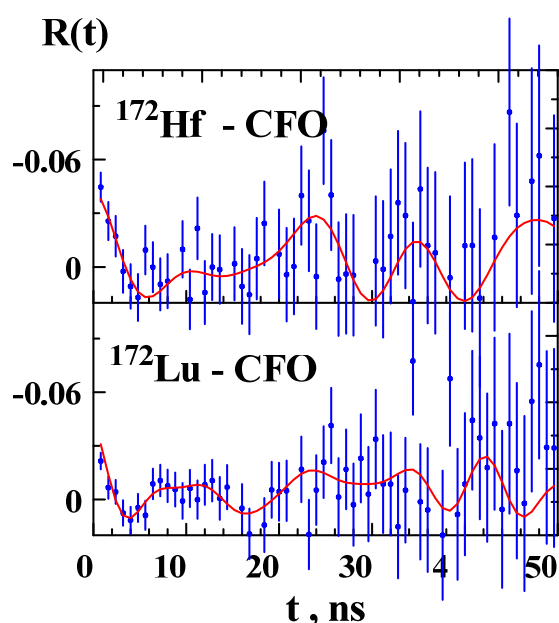


Fig. 6. The ^{172}Yb PAC spectra in CoFe_2O_4 ferrite measured at 298 K, after the introduction of ^{172}Hf (above) and ^{172}Lu (below) into the sample

It should be noted that below the Curie temperature, the hyperfine field is a mix of magnetic dipole and electric quadrupole fields. To reveal more clearly the influence of dopant ions (Hf and Lu) on the electric quadrupole interaction (directly related to the position of the dopant ion in the crystal lattice), we carried out measurements above the Curie temperature (1000 K). Table 2 shows the obtained quadrupole frequency, electric field gradient, and relative site occupancy parameters for ^{172}Yb in CoFe_2O_4 (CFO), after introducing ^{172}Lu or ^{172}Hf into the sample.

The observed difference in the parameters can be explained by the assumption that the ^{3+}Lu and ^{4+}Hf ions are distributed differently when introduced into two non-equivalent lattice sites: in addition to the different charges of these ions, the lutetium ion has a larger ionic radius.

Basically, the post-effects of nuclear decay may influence the local environment of ^{172}Yb

in CFOs. However, in crystalline phases, these effects usually have no significant influence on the HFI parameters.

4. Conclusions

Using the Mössbauer spectroscopy on ^{57}Fe nuclei, we studied the hyperfine interactions in CoFe_2O_4 ferrites doped with Hf, Lu, and Yb (1 wt. %). It was shown that doping with Lu and Yb leads to the formation of additional non-magnetic phases (Lu – 11 % and Yb – 23.4 %). In the ferrite doped with hafnium Hf, no additional phase was observed. A significant change in the HFI parameters was revealed for ^{57}Fe in the octahedral sites of CoFe_2O_4 ferrites doped with Hf, Yb, or Lu, respectively. However, the relative influence of the impurity on ^{57}Fe nuclei in the tetrahedral sites was insignificant. The formation of a new phase in the case of Yb and Lu can be attributed to the fact that the ionic radii of Yb and Lu are about 14–13 % larger than the ionic radius of Hf.

The parameters of hyperfine interactions on ^{172}Yb nuclei in CoFe_2O_4 ferrites were obtained by the PAC method. The ^{172}Yb ions were introduced (10^{-7} – 10^{-8} wt. %) into the samples in two ways: via ^{172}Hf ($^{172}\text{Hf} \rightarrow ^{172}\text{Lu} \rightarrow ^{172}\text{Yb}$ chain) or via the parent ^{172}Lu . A significant difference in the HFI parameters for these two cases was revealed. The difference in the HFI parameters persisted in the temperature range of 300–1000 K. The observed difference is due to the different distribution of ^{3+}Lu and ^{4+}Hf over the lattice sites due to different charges and ionic radii of these ions.

Notably, the different effects of Hf and Lu on the parameters (electric field gradient, magnetic field, and isomer shift) of the HFI in the samples were revealed by both MS and PAC methods, irrespective of the amount of the dopant. Specifically, according to MS, Hf, and Lu did not cause significant changes in the HFI parameters in the tetrahedral sites (“Sextet 1”),

Table 2. The parameters of the hyperfine interactions (above the Curie temperature) for ^{172}Yb upon the introduction of ^{172}Lu or ^{172}Hf into CFOs: quadrupole frequency ω_Q , electric field gradient V_{zz} , and relative site occupation f

	ω_Q , Мрад/с	V_{zz} , 10^{21} В/м ²	$f(\omega_1)$, %	ω_Q , Мрад/с	V_{zz} , 10^{21} В/м ²	$f(\omega_2)$, %
$^{172}\text{Hf}(^{172}\text{Lu}(^{172}\text{Yb}))$	220(15)	10.12(69)	65(8)	60(8)	2.76(37)	35(7)
$^{172}\text{Lu}(^{172}\text{Yb})$	231(5)	10.63(56)	55(7)	108(9)	4.97(41)	45(8)

but had significantly different effects on the same parameters in the octahedral sites ("Sextet 2"). A similar situation was observed using the PAC method: substitution of Hf by Lu did not reveal a change in the HFI parameters in one of the sites (according to [15], it is the octahedral site), but indicated a significant difference (see Table 2) in the other site.

Contribution of the authors

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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